U.S. Department of Energy

Mixed-Analyte Performance Evaluation Program (MAPEP)

MAPEP TEST SESSION 12

CLOSING DATE: October 27, 2004 24:00 (MST)

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Radioactive Decay Correction Date for All Radiological Samples:
May 1, 2004, 12:00 Mountain Standard Time (MST).

PLEASE READ ALL INSTRUCTIONS

CAREFULLY BEFORE ANALYZING SAMPLE

IMMEDIATELY UPON RECEIPT OF SAMPLES
CHECK FOR BREAKAGE AND SHIPPING ERRORS;
SAMPLE REPLACEMENT TAKES AT LEAST
2 TO 3 DAYS

INSTRUCTIONS FOR MAPEP TEST SESSION 12

1. MAPEP PERFORMANCE EVALUATION (PE) SAMPLES.

Water Sample:
Domestic laboratories performing radiological/inorganic and organic analyses may receive as many as three one-liter sample bottles (MaW – mixed analyte radiological and stable inorganic water, GrW – Gross alpha/beta water, and OrW – semi-volatile organics water sample). If requesting the semi-volatile organics PE sample, the organic shipment should follow the radiological/inorganic shipment by about one week. **It is critical that radiological and stable inorganic analyses utilize sample from ONLY the bottle marked for mixed analyte radiological and stable inorganic analyses (MaW). Gross alpha/beta analyses must utilize sample from ONLY the bottle marked for gross alpha/beta analyses (GrW). Organic analyses must utilize sample from ONLY the bottle marked for organic analyses (OrW)**. Failure to utilize the appropriate sample bottle will yield incorrect results.

Note: the semi-volatile organics water (OrW) will not be distributed for test session 12. It will be available for the next test session.

Soil Sample:
Most laboratories performing radiological, stable inorganic, and/or organic analyses will receive one ~300 gram sample jar of soil. Laboratories that requested extra soil will receive more than one jar. The soil contains all analytes (radiological, stable inorganic, and semi-volatile organics) and is labeled as a mixed analyte soil (MaS).

Air Filters:
Air filters are spiked with radiological constituents only, i.e., they are not mixed analyte samples. Laboratories performing radiological and gross alpha/beta air filter analyses will receive one to three filter packets, dependent upon the analyses performed. Filters labeled RdF are radiological air filters with multiple radionuclide determinations required. A filter labeled GrF is for gross alpha/beta analyses only. Air filters that require multiple radionuclide determinations (RdF) will come in two filter packets. Each filter packet contains an identically spiked air filter sandwiched between upper and lower non-spiked filters. The spiked side of the middle filter is placed in the packet facing "up" toward the label. The second air filter may be used for screening, the non-sequential determination of Sr-90, gamma-ray spectrometry, or other analytical procedures as needed.

The gross alpha/beta air filter will come in one filter packet containing one filter (GrF). The spiked side of the filter is placed in the packet facing "up" toward the label. For gross calibration information, the gross alpha/beta filters are spiked with Th-230 and Sr-90. The RdF and GrF filters are not marked so carefully note the spiked filters and their orientation before removing them from the packets.

2. SAMPLE DESCRIPTIONS.

Sample descriptions for the water, soil, and air filters associated with this study are found toward the end of these instructions. Analyze the sample for those analytes that comprise your routine function or constitute your compliance requirements.

3. REPORTING ANALYTICAL RESULTS.

REPORTING RADIOLOGICAL ANALYTES:
Radiological analyses should report results for only the analytes listed on the sample description. Other analytes may be detectable but will not be evaluated. Conversely, some of the radiological analytes listed on the sample description may not be detected. Report the actual results obtained for all analyses performed, including negative numbers, even if the radionuclide was not detected (i.e., do not report results as "Less Than" or "Not Detected"). The result and total propagated uncertainty are required for sensitivity determinations and false positive testing. Failure to report results for requested analyses may result in a "Not Acceptable" performance evaluation if the analysis is within the scope of your routine function or contractual obligations. Report all results in Becquerels per unit, i.e., Bq/L (water), Bq/kg (soil), and Bq/sample (filter).

REPORTING STABLE INORGANIC ANALYTES:
Stable inorganic analyses should report results for only the analytes listed on the sample description. Other analytes may be detectable but will not be evaluated. Conversely, some of the stable inorganic analytes listed on the sample description may not be detected. Report the actual results obtained, or if applicable, the detection limit value. "Less Than" ('<') with an established Detection Limit is acceptable for reporting results for stable inorganic analytes. **DO NOT USE 0.00 (zero) as a reportable value.** Results reported as zeros will be flagged as "Not Acceptable". Total uranium, uranium-238, and uranium-235 can be reported when utilizing mass spectrometric techniques under the reporting section for stable inorganic analytes. Report all results based on a sample weight in milligrams per unit, i.e., mg/L (water), mg/kg (soil), and mg/sample (filter; for mass spec uranium results only).

REPORTING SEMIVOLATILE ORGANIC ANALYTES:
Organic analyses should report results for only the detectable analytes from the targeted organic classes. Report all results in micrograms per unit, i.e., μg/L (water) and μg/kg (soil). **DO NOT USE CLP reporting flags (U, J, etc.). DO NOT USE 0.00 (zero) as a reportable value.**

FOR ALL ANALYTES:
You are required to report only one result for each appropriate analyte. If the reported result is actually a mean of several replicate analyses, the reported uncertainty should also be the mean of the individual uncertainties. Do not propagate the individual uncertainties for replicate measurements. For example, assume three replicate analyses provided the following results and individual uncertainties: 101 +/- 12, 108 +/- 15, 110 +/- 16. The mean result is (101+108+110)/3=106 and the mean individual uncertainty is (12+15+16)/3=14. The reported result and uncertainty is 106 +/- 14. The reported total uncertainty should be at

one standard deviation. If propagated uncertainties are not currently available for stable inorganic or organic analyses, you may report laboratory control sample (LCS) or surrogate spike data (see the MAPEP Handbook). Propagated uncertainties are strongly encouraged. Please also ensure that the Method Code is entered correctly for each reported result. Method Codes are used in proficiency testing and an inappropriate Method Code may result in a "Not Acceptable" performance evaluation.

Report your results electronically via the MAPEP World Wide Web application at **http://mapep.inel.gov/.** Login information, including user ID and password, were previously emailed to the MAPEP point of contact (POC) for your laboratory. Please ensure that your lab code, points of contact, addresses, and NRC license information are entered correctly in the data entry program. The shipping distribution list and correspondence mailing list will utilize the address and POC information exactly as you enter it here. You are a U.S. Federal Laboratory only if your employees are federal government workers (i.e., EPA, USGS, NRC, etc.). If you are a primary contractor for a DOE National Laboratory you may have a DOE exemption and, if so, enter your DOE contract number.

4. ADDITIONAL INFORMATION.

4.1 The laboratory may choose the analytical method.

4.2 The amount of sample is limited. The laboratory should use the maximum specific activities and concentration ranges listed on the sample description to select the optimum amount of sample for each analysis to ensure that sufficient sample is available for all of the analyses.

4.3 Excess sample or residues shall not be returned to RESL. Do not initiate analysis of the sample if approved waste treatment, storage, or disposal options are not available.

4.4 The reference date for radioactive decay correction for all radiological analyses is **May 1, 2004, 12:00 Mountain Standard Time (MST)**. Sample-holding time is based upon the RECEIPT date of the sample by the participating laboratory.

4.5 **Results are due by October 27, 2004 24:00 (MST).** Late results will not be included in the final report.

4.6 Please address any questions to the appropriate point of contact:

Jim Dahlgran (208-526-6243, dahlgrjr@inel.gov): data entry and organic analyses;
Leon Jensen (208-526-4591, jensenll@inel.gov): stable inorganic analyses;
David Sill (208-526-8031, sillds@inel.gov): radiological analyses.

MAPEP-04-MaW12 WATER SAMPLE DESCRIPTION

The analytes for the MAPEP water, and their maximum specific activities and concentration ranges, are listed in the following tables. Each radiological/stable inorganic sample contains approximately one liter of 5% (v/v) nitric acid in water.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 15 Bq/L	^{57}Co, ^{134}Cs, ^{137}Cs, ^{55}Fe, ^{63}Ni, ^{54}Mn, ^{65}Zn, ^{60}Co	< 2000 Bq/L
^{90}Sr, ^{99}Tc	< 100 Bq/L	^{3}H	<1000 Bq/L

NOTE: The ^{234}U and ^{238}U isotopes may not be in equilibrium. Some of the radionuclides listed on the sample description may not be detected, but if included in your sample analyses, the result and total propagated uncertainty must be reported for sensitivity evaluation and/or false positive testing. The reference date for decay correction is May 1, 2004, 12:00 MST.

STABLE INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration Range	Analyte	Concentration Range
Ag, As, Cr (Total), Ni, Pb, Sb, V	0.01 – 4.9 mg/L	Tl, Zn	0.01 – 10 mg/L
Be, Cd, Se	0.01 – 0.9 mg/L	Ba, Cu	0.1 - 95 mg/L

NOTE: Some of the stable inorganic constituents listed in the above table may not be present in the sample. Laboratories should report results and associated uncertainties for those constituents quantitated above the minimum concentration range listed for that analyte. For sensitivity evaluation and/or false positive testing, the actual analytical or detection limit values should be reported for those constituents with results found to be less than the lower concentration range. Failure to report analytical results as instructed may result in a false positive or false negative performance evaluation.

SEMI-VOLATILE ORGANIC SAMPLE DESCRIPTION

The semi-volatile organics PE water sample (OrW) will NOT be distributed for test session 12. It will be available for the next test session.

MAPEP-04-MaS12 SOIL SAMPLE DESCRIPTION

The analytes for the MAPEP soil, and their maximum specific activities and concentration ranges, are listed in the following tables. Most participants will receive a single sample containing approximately 300 grams of soil.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{57}Co, ^{134}Cs, ^{137}Cs, ^{54}Mn, ^{65}Zn, ^{60}Co, ^{40}K	< 4000 Bq/kg	^{55}Fe, ^{63}Ni	< 2000 Bq/kg
^{90}Sr, ^{99}Tc	< 1000 Bq/kg	^{241}Am, ^{238}Pu, ^{239}Pu ^{234}U, ^{238}U	< 300 Bq/kg

NOTE: The ^{234}U and ^{238}U isotopes may NOT be in equilibrium. Some of the radionuclides listed on the sample description may not be detected, but if included in your sample analyses, the result and total propagated uncertainty must be reported for sensitivity evaluation and/or false positive testing. Tc-99 is a new analyte added to the soil for this test session. The reference date for decay correction is May 1, 2004, 12:00 MST.

STABLE INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration Range	Analyte	Concentration Range
Tl, Ni, V, Sb, Zn	10 - 400 mg/kg	Ba	100 - 1800 mg/kg
Ag, As, Cr (Total), Pb	5 – 95 mg/kg	Be	5 - 50 mg/kg
Cd, Se	1 – 19 mg/kg		

NOTE: Some of the stable inorganic constituents listed in the above table may not be present in the sample. Laboratories should report results and associated uncertainties for those constituents quantitated above the minimum concentration range listed for that analyte. For sensitivity evaluation and/or false positive testing, the actual analytical or detection limit values should be reported for those constituents with results found to be less than the lower concentration range. Failure to report analytical results as instructed may result in a false positive or false negative performance evaluation.

SEMI-VOLATILE ORGANIC SAMPLE DESCRIPTION

Analyte Class	Concentration Range	Analyte Class	Concentration Range
Phthalate Esters	< 1100 ug/kg	Polynuclear Aromatics	< 1200 ug/kg
Phenols	< 1000 ug/kg	Nitroaromatics	< 1100 ug/kg
Chlorinated Pesticides	< 20 ug/kg	Chlorinated Hydrocarbons	< 1000 ug/kg
	ug = micrograms	kg = kilograms	

NOTE: Sample-holding time is based upon the RECEIPT date of the sample by the participating laboratory.

"MAPEP samples are analytical standards or a "product" generated for the purpose of securing and evaluating analytical services; they are not hazardous waste and they are not samples of hazardous waste... Thus, a laboratory participating in the MAPEP is in the process of establishing its eligibility and credentials to do DOE analytical work. It follows, therefore, that the laboratory is the "generator" of the waste resulting when the samples and the resulting residues are to be discarded." (MEMORANDUM OCC-95-189, Office of Chief Counsel, October 16, 1995)

MAPEP-04-RdF12 RADIOLOGICAL AIR FILTER SAMPLE DESCRIPTION

The analytes for the MAPEP radiological air filters and their maximum specific activities are listed in the following table. Each filter packet contains an identically spiked filter sandwiched between upper and lower non-spiked filters. The spiked side of the middle filter is placed in the packet facing "up" toward the label**.**

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 2 Bq/sample	^{57}Co, ^{134}Cs, ^{137}Cs, ^{54}Mn, ^{65}Zn, ^{60}Co	< 6 Bq/sample
^{90}Sr	< 4 Bq/sample		

MAPEP-04-GrW12 GROSS ALPHA/BETA WATER SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP gross alpha/beta water is listed in the following table. Each sample contains approximately one liter of 5% (v/v) nitric acid in water.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Concentration Range
Gross Alpha (Th-230)	< 2 Bq/L
Gross Beta (Sr-90)	< 3 Bq/L

MAPEP-04-GrF12 GROSS ALPHA/BETA AIR FILTER SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP gross alpha/beta air filter is listed in the following table. The filter packet contains one filter. The spiked side of the filter is placed in the packet facing "up" toward the label.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Concentration Range
Gross Alpha (Th-230)	< 2 Bq/sample
Gross Beta (Sr-90)	< 3 Bq/sample

MAPEP Sample Semi-Volatile Organic Target Compounds

Soil (MaS) and water (OrW) samples may contain any of the following compounds.

MAPEP Target Analyte List

Phenols

4-Chloro-3-methylphenol
2-Chlorophenol
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
4,6-Dinitro-2-methylphenol
2-Methylphenol
4-Methylphenol
3-Methylphenol
2-Nitrophenol
4-Nitrophenol
Pentachlorophenol
Phenol
2,3,4,6-Tetrachlorophenol
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
Dinoseb

Chlorinated Hydrocarbons

2-Chloronaphthalene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
1,2,4,5-Tetrachlorobenzene
1,2,4-Trichlorobenzene
Pentachlorobenzene
Pentachloronitrobenzene
4-Chloroanaline

Other

o-Toluidene
Benzyl alchohol
Dibenzofuran
2-Naphthylamine
Analine
1,4-phenylenediamine

Nitroaromatics Cyclic Ketones

Nitrobenzene
1,3-Dinitrobenzene
1,2-Dinitrobenzene
1,4-Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
2-Nitroanaline
3-Nitroanaline
4-Nitroanaline
Isophorone
1,4-naphthoquinone

Phthalate Esters

Dimethylphthalate
Diethylphthalate
Di-n-butylphthalate
Butylbenzylphthalate
Bis(2-ethylhexyl)phthalate
Di-n-octylphthalate

PAHs

2-methylnaphthalene
Naphthalene
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-c,d)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

Chlorinated Pesticides

alpha-BHC
beta-BHC
Lindane
delta-BHC
Heptachlor
Aldrin
Heptachlor epoxide
Endosulfan I
4,4'-DDE
Dieldrin
Endrin
4,4'-DDD
Endosulfan II
4,4'-DDT
Endrin Aldehyde
Endosulfan Sulfate
Endrin Ketone
Methoxychlor

MATERIAL SAFETY DATA SHEETS ARE ENCLOSED

END OF INSTRUCTIONS